Pearson Edexcel IAS Chemistry

WCH13

REVISION GUIDE

This guide includes details about the core practicals for A-level chemistry. It also contains information about other experiments that often occur in A-level examinations. You may be asked to describe these experiments in details or be asked about reasons for doing individual steps.

You may be asked about other unfamiliar experiments but these will be using the skills and techniques that are described in the following experiments.

Safety and hazards

Irritant - dilute acid and alkalis- wear googles
Corrosive- stronger acids and alkalis wear goggles
Flammable – keep away from naked flames
Toxic – wear gloves- avoid skin contact- wash hands after use
Oxidising- Keep away from flammable / easily oxidised materials

Hazardous substances in low concentrations or amounts will not pose the same risks as the pure substance.

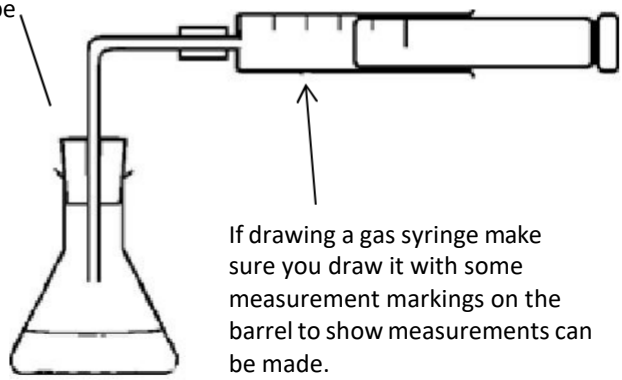
Measuring gas volumes

Using a gas syringe

Gas syringes can be used for a variety of experiments where the volume of a gas is measured, possibly to work out moles of gas or to follow reaction rates.

Potential errors in using a gas syringe •gas escapes before bung inserted •syringe sticks
• some gases like carbon dioxide or sulphur dioxide are soluble in water so the true amount of gas is not measured.

Make sure you don't leave gaps in your diagram where gas could escape

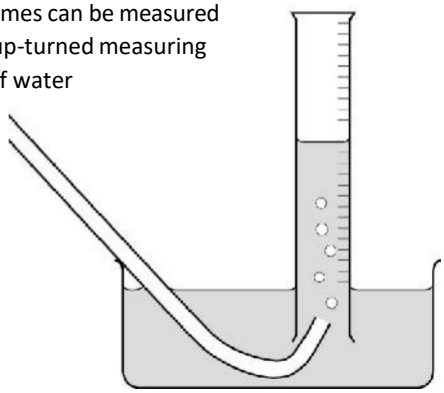


If drawing a gas syringe make sure you draw it with some measurement markings on the barrel to show measurements can be made.

The volume of a gas depends on pressure and temperature so when recording volume it is important to note down the temperature and pressure of the room.

Moles of gas can be calculated from gas volume (and temperature and pressure) using ideal gas equation PV = nRT or using the molar gas volume (1mol gas =24dm^3 at room temperature and pressure

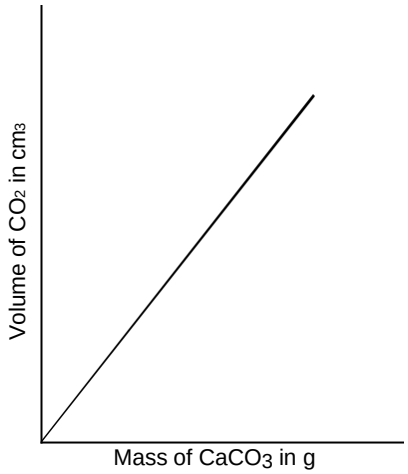
Alternatively gas volumes can be measured 'over water' with an up-turned measuring cylinder in a trough of water



Core Practical 1:Measure the molar volume of a gas

Detailed method

1. Measure 30 cm_3 of 1 mol dm^{-3} ethanoic acid and transfer to a conical flask.
2. Attach conical flask to gas syringe or use collection over water method (see previous page)
3. Measure the mass of a weighing bottle with approximately 0.05 g of calcium carbonate
4. Add the calcium carbonate to the conical flask- quickly resealing the bung so no gas escapes
5. Measure the final total volume of gas
6. Reweigh the empty weighing bottle test tube from step 3
7. Repeat the experiment several more times, increasing the mass of calcium carbonate by about 0.05 g each time.



Analysis

From the graph read the volume of CO_2 given off with 0.25 g $CaCO_3$

Work out the moles of $CaCO_3$ in 0.25g = 0.25/100.1 = 2.5 x 10^{-3} Assume the moles of CO_2 = moles of $CaCO_3$

Work out molar volume of CO_2 = volume of CO_2/ moles of CO_2

Method for using a gas syringe to calculate the *Mr* of propanone

1. Extract 0.20 cm^3 of propanone into a hypodermic syringe and then measure the mass of this syringe
2. using hand protection, remove a gas syringe from the oven and note the volume of air already in the barrel – about 5 cm^3.
3. inject the propanone through the self-seal cap into the barrel. The plunger will move straight away.
4. Put the gas syringe back into the oven.
5. Measure the mass of the empty hypodermic syringe immediately.
6. After a few minutes measure the volume of the gas in the gas syringe, record the temperature of the oven shelf and the pressure of the room.

Example 1 : 0.150g of a volatile liquid was injected into a sealed gas syringe. The gas syringe was placed in an oven at 70^oC at a pressure of 100kPa and a volume of 80cm^3 was measured. Calculate the *Mr* of the volatile liquid (R = 8.31)

moles = PV/RT

= 100 000 x 0.00008 / (8.31 x 343)

= 0.00281 mol

100 kPa = 100 000 Pa

80 cm^3 = 0.00008 m^3

Mr = mass/moles

= 0.15 / 0.00281

= 53.4 g mol^{-1}

Heating in a crucible

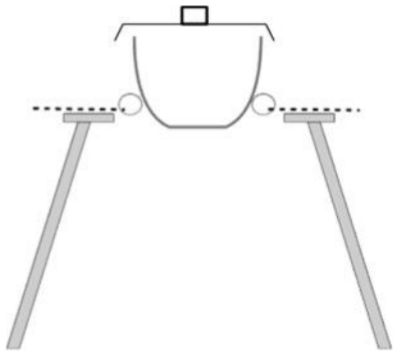
This method could be used for measuring mass loss in various thermal decomposition reactions and also for mass gain when reacting magnesium in oxygen.

The water of crystallisation in calcium sulfate crystals can be removed as water vapour by heating as shown in the following equation. $CaSO_4.xH_2O(s) \rightarrow CaSO_4(s) + xH_2O(g)$

Method.

- Weigh an empty clean dry crucible and lid .
- Add 2g of hydrated calcium sulfate to the crucible and weigh again
- Heat strongly with a Bunsen for a couple of minutes
- Allow to cool
- Weigh the crucible and contents again
- Heat crucible again and reweigh until you reach a constant mass (do this to ensure reaction is complete).

The lid improves the accuracy of the experiment as it prevents loss of solid from the crucible but should be loose fitting to allow gas to escape.



Large amounts of hydrated calcium sulfate, such as 50g, should not be used in this experiment as the decomposition is likely to be incomplete.

The crucible needs to be dry otherwise a wet crucible would give an inaccurate result. It would cause mass loss to be too large as the water would be lost when heating.

Small amounts of the solid , such as 0.100 g, should **not** be used in this experiment as the percentage uncertainties in weighing will be too high.

Example 2. 3.51 g of hydrated zinc sulfate were heated and 1.97 g of anhydrous zinc sulfate were obtained. Use these data to calculate the value of the integer *x* in $ZnSO_4.xH_2O$

Calculate the mass of $H_2O = 3.51 - 1.97 = 1.54g$

Calculate moles of $ZnSO_4 = \frac{1.97}{161.5} = 0.0122$

Calculate moles of $H_2O = \frac{1.54}{18} = 0.085$

Calculate ratio of mole of $ZnSO_4$ to $H_2O = \frac{0.0122}{0.0122} = 1$: $\frac{0.085}{0.0122} = 7$

X = 7

CORE PRACTICAL 2: To determine the enthalpy change of a reaction using Hess's Law

Measuring the Enthalpy Change for a Reaction

Experimentally Calorimetric method

For a reaction in solution we use the following equation

energy change = mass of solution x heat capacity x temperature change

Q (J) = m (g) x c_p (J $g^{-1}K^{-1}$) x ΔT (K)

This equation will only give the energy for the actual quantities used. Normally this value is converted into the energy change per mole of one of the reactants. (The enthalpy change of reaction, ΔH)

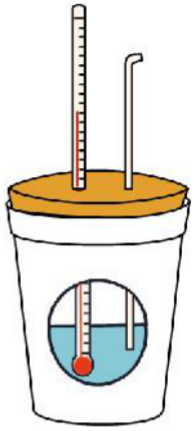
Calorimetric method

One type of experiment is one in which substances are mixed in an insulated container and the temperature rise measured.

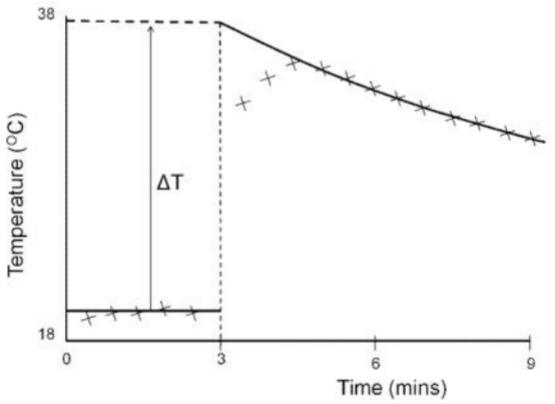
This could be a solid dissolving or reacting in a solution or it could be two solutions reacting together

General method

- washes the equipment (cup and pipettes etc) with the solutions to be used
- dry the cup after washing
- put polystyrene cup in a beaker **for insulation and support**
- Measure out desired volumes of solutions with volumetric pipettes and transfer to insulated cup
- clamp thermometer into place making sure the thermometer bulb is immersed in solution
- measure the initial temperatures of the solution or both solutions if 2 are used. Do this every minute for 2-3 minutes
- At minute 3 transfer second reagent to cup. If a solid reagent is used then add the solution to the cup first and then add the solid weighed out on a balance.
- If using a solid reagent then use 'before and after' weighing method
- stirs mixture **(ensures that all of the solution is at the same temperature)**
- Record temperature every minute after addition for several minutes



If the reaction is slow then the exact temperature rise can be difficult to obtain as cooling occurs simultaneously with the reaction



To counteract this we take readings at regular time intervals and extrapolate the temperature curve/line back to the time the reactants were added together.

We also take the temperature of the reactants for a few minutes before they are added together to get a better average temperature. If the two reactants are solutions then the temperature of both solutions need to be measured before addition and an average temperature is used.

Errors in this method

- energy transfer from surroundings (usually loss)
- approximation in specific heat capacity of solution. The method assumes all solutions have the heat capacity of water.
- neglecting the specific heat capacity of the calorimeter- we ignore any energy absorbed by the apparatus.
- reaction or dissolving may be incomplete or slow.
- Density of solution is taken to be the same as water.

Read question carefully. It may be necessary to

describe:

- Method
- Drawing of graph with extrapolation
- Description of the calculation

Calculating the enthalpy change of reaction, ΔH_r from experimental data

General method

1. Using **$q = m \times c_p \times \Delta T$** calculate energy change for quantities used
2. Work out the moles of the reactants used
3. Divide q by the number of moles of the reactant not in excess to give **ΔH**
4. Add a sign and unit (divide by a thousand to convert $Jmol^{-1}$ to $kJmol^{-1}$

The heat capacity of water is 4.18 J $g^{-1}K^{-1}$. In any reaction where the reactants are dissolved in water we assume that the heat capacity is the same as pure water.

Also assume that the solutions have the density of water, which is 1g cm^{-3}. Eg 25cm^3 will weigh 25 g

Example 3. Calculate the enthalpy change of reaction for the reaction where 25.0cm^3 of 0.20M copper sulfate was reacted with 0.01mol (excess of zinc). The temperature increased 7.0^oC .

Step 1: Calculate the energy change for the amount of reactants in the calorimeter.

$Q = m \times c_p \times \Delta T$

$Q = 25 \times 4.18 \times 7$

$Q = 731.5$ **J**

Note the mass is the mass of the copper sulfate solution only. Do not include mass of zinc powder.

Step 2 : calculate the number of moles of the reactant not in excess.

moles of $CuSO_4$ = conc x vol
= 0.2 x 25/1000
= 0.005 mol

If you are not told what is in excess, then you need to work out the moles of both reactants and work out using the balanced equation which one is in excess.

Step 3 : calculate the enthalpy change per mole, ΔH (the enthalpy change of reaction) ΔH = Q/ no of moles
= 731.5/0.005
= 146300 **J mol^{-1}**
= 146 **kJ mol^{-1} to 3 sf**

Finally add in the sign to represent the energy change: if temp increases the reaction is exothermic and is given a minus sign e.g. **–146 kJ mol^{-1}**

Remember in these questions: **sign, unit**

Example 4. 25.0cm^3 of 2.0 mol dm^{-3} HCl was neutralised by 25.0cm^3 of 2.0 mol dm^{-3} NaOH. The temperature increased 13.5^oC. Calculate the enthalpy change per mole of HCl?

Step 1: Calculate the energy change for the amount of reactants in the test tube.

$Q = m \times c_p \times \Delta T$

$Q = 50 \times 4.18 \times 13.5$

$Q = 2821.5$ **J**

Note the mass equals the mass of acid + the mass of alkali, as they are both solutions.

Step 2 : calculate the number of moles of the HCl.

moles of HCl = conc x vol
= 2 x 25/1000
= 0. 05 mol

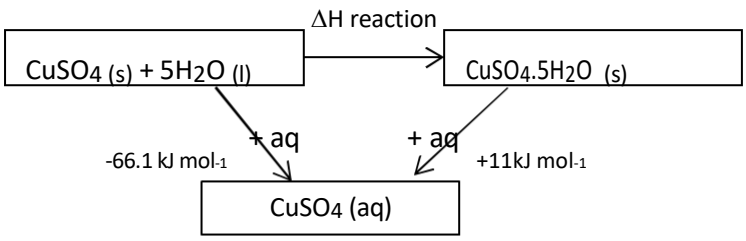
Step 3 : calculate ΔH the enthalpy change per mole which can be called the enthalpy change of neutralisation

ΔH = Q/ no of moles
= 2821.5/0.05
= 56430 **J mol^{-1}**
= -56.4 **kJ mol^{-1} to 3 sf**

Exothermic and so is given a minus sign

Remember in these questions: **sign, unit,**

Often Hess's law cycles are used to measure the enthalpy change for a reaction that cannot be measured directly by experiments. Instead alternative reactions are carried out that can be measured experimentally.



ΔH reaction + 11kJmol^{-1} = -66.1 kJ mol^{-1}

ΔH reaction= -66.1 - 11

= -77.1 kJ mol^{-1}

This Hess's law is used to work out the enthalpy change to form a hydrated salt from an anhydrous salt.

This cannot be done experimentally because it is impossible to add the exact amount of water without the solid dissolving and it is not easy to measure the temperature change of a solid.

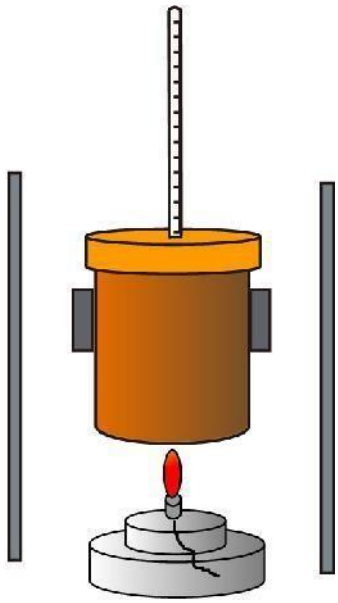
Instead both salts are dissolved in excess water to form a solution of copper sulfate. The temperature changes can be measured for these reactions.

Detailed method for measuring enthalpy change of solution of anhydrous copper(II) sulfate

1. Weigh out between 3.90 g and 4.10 g of anhydrous copper(II) sulfate in a dry weighing bottle. The precise mass should be recorded.
2. Using a volumetric pipette, place 25 cm^3 of deionised water into a polystyrene cup and record its temperature at the beginning (t=0), start the timer and then record the temperature again every minute, stirring the liquid continuously.
3. At the fourth minute, add the powdered anhydrous copper(II) sulfate rapidly to the water in the polystyrene cup and continue to stir, but do not record the temperature.
4. Reweigh the empty weighing bottle
5. At the fifth minute and for every minute up to 15 minutes, stir and record the temperature of the solution in the polystyrene cup.
6. Plot a graph of temperature (on the y-axis) against time. Draw two separate best fit lines; one, which joins the points before the addition, and one, which joins the points after the addition, extrapolating both lines to the fourth minute.
7. Use your graph to determine the temperature change at the fourth minute, which theoretically should have occurred immediately on addition of the solid.
8. Using **$q = m \times c_p \times \Delta T$** calculate energy change
   = 20 x 4.18 **$\times \Delta T$**
9. Calculate $\Delta H_{solution}$ by dividing q by number of moles of anhydrous copper(II) sulfate in mass added

The above method is then repeated using hydrated copper sulfate. The two $\Delta H_{solution}$ can then be used to calculate the ΔH for the enthalpy change of forming a hydrated salt as in the example above

Measuring Enthalpies of Combustion using Flame Calorimetry



Enthalpies of combustion can be calculated by using calorimetry. Generally the fuel is burnt and the flame is used to heat up water in a metal cup.
Need to measure

- mass of spirit burner before and after
- Temperature change of water
- Volume of water in cup

Errors in this method

- energy losses from calorimeter
- Incomplete combustion of fuel
- Incomplete transfer of energy
- Evaporation of fuel after weighing
- Heat capacity of calorimeter not included
- Measurements not carried out under standard conditions as H_2O is gas, not liquid, in this experiment

Example 5. Calculate the enthalpy change of combustion for the reaction where 0.65g of propan-1-ol was completely combusted and used to heat up 150g of water from 20.1 to 45.5^{o}C

Step 1: Calculate the energy change used to heat up the water.

$Q = m \times c_p \times \Delta T$

$Q = 150 \times 4.18 \times 25.4$

$Q = 15925.8$ **J**

Note the mass in this equation is the mass of water in the calorimeter and not the alcohol

Step 2 : calculate the number of moles of alcohol combusted.

moles of propan-1-ol = mass/ *M*r
= 0.65 / 60
= 0.01083 mol

Step 3 : calculate the enthalpy change per mole which is called ΔcH (the enthalpy change of combustion)

ΔH = Q/ no of moles
= 15925.8/0.01083
= 1470073 **J mol^{-1}**
= 1470 **kJ mol^{-1} to 3 sf**

Finally add in the sign to represent the energy change: if temp increases the reaction is exothermic and is given a minus sign eg **–1470 kJ mol^{-1}**

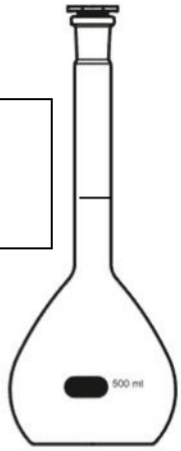
Remember in these questions: **sign, unit**

Core practical 3+4. Make up a volumetric solution and carry out a simple acid–base titration

Making a solution

- Weigh the sample bottle containing the required mass of solid on a 2 dp balance
- Transfer to beaker
- Reweigh empty sample bottle
- Record the difference in mass

Alternatively the known mass of solid in the weighing bottle could be transferred to beaker, washed and washings added to the beaker.



• Add 100cm^3 of distilled water to the beaker. Use a glass rod to stir to help dissolve the solid.

•Sometimes the substance may not dissolve well in cold water so the beaker and its contents could be heated gently until all the solid had dissolved.

- Pour solution into a 250cm^3 graduated flask via a funnel.
- Rinse beaker and funnel and add washings from the beaker and glass rod to the volumetric flask.

• make up to the mark with distilled water using a dropping pipette for last few drops.

• Invert flask several times to ensure uniform solution.

Remember to fill so the bottom of the meniscus sits on the line on the neck of the flask. With dark liquids like potassium manganate it can be difficult to see the meniscus.

Shake the volumetric flask thoroughly to ensure a uniform concentration

Graduated/volumetric flask

A graduated flask has one mark on the neck which the level to fill to get the accurate volume. Do not heat or put hot solutions in the volumetric flask because the heat would cause the flask to expand and the volume would then be incorrect.

Measuring mass accurately:
In many experiments the best method for measuring mass is

1. Measure mass on 2 or 3d.p. balance of a weighing bottle with the required quantity of solid in it
2. Empty mass into reaction vessel/flask
3. Reweigh the now empty weighing bottle
4. Subtract the mass of the empty weighing bottle from the first reading to give exact of mass actually added.

Dilutions

Diluting a solution

•Pipette 25cm^3 of original solution into a 250cm^3 volumetric flask

•make up to the mark with distilled water using a dropping pipette for last few drops.

• Invert flask several times to ensure uniform solution.

Using a volumetric pipette is more accurate than a measuring cylinder because it has a smaller uncertainty

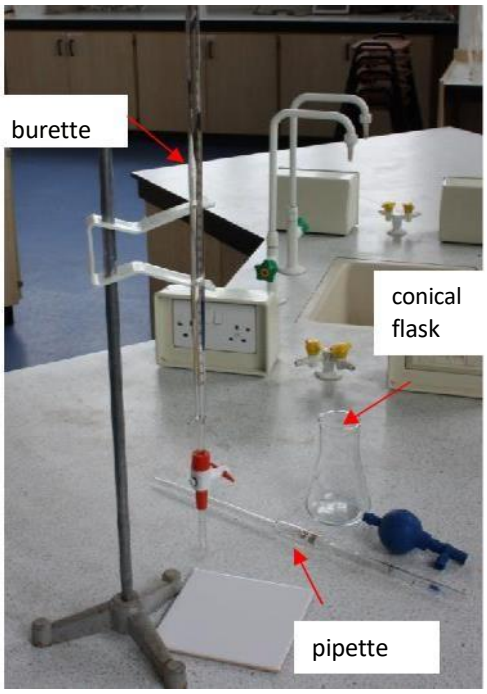
Use a teat pipette to make up to the mark in volumetric flask to ensure volume of solution accurately measured and one doesn't go over the line

Titrations **Core Practical 3+4: Make up a volumetric solution and carry out a simple acid–base titration**

General Method
- **rinse equipment** (burette with acid, pipette with alkali, conical flask with distilled water)
- **pipette 25 cm^3 of alkali into conical flask**
- **touch surface of alkali with pipette** (to ensure correct amount is added)
- **adds acid solution from burette**
- **make sure the jet space** in the burette **is filled** with acid
- add **a few drops of indicator** and refer to colour change at end point
- phenolphthalein [pink (alkali) to colourless (acid): end point pink colour just disappears] [use if NaOH is used]
- methyl orange [yellow (alkali) to red (acid): end point orange] [use if HCl is used]
- use a white tile underneath the flask to help observe the colour change
- add acid to alkali whilst **swirling the mixture** and **add acid drop wise at end point**
- **note burette reading** before and after addition of acid
- **repeats titration** until **at least 2 concordant results** are obtained- two readings within 0.1 of each other

Titrations are done often to find out the concentration of one substance by reacting it with another substance of known concentration.

They are often done with neutralisation reactions, but can be done with redox reactions.



One substance (generally the one we don't know the concentration) is put in the conical flask. It is measured using a volumetric pipette.
The other substance is placed in the burette

However, the standard phrase: **titrate solution A with solution B** means that A should be in the conical flask and B should be in the burette.

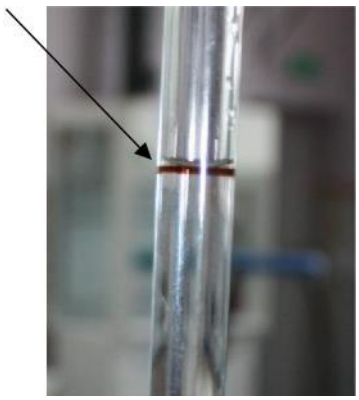
A conical flask is used in preference to a beaker because it is easier to swirl the mixture in a conical flask without spilling the contents.

Detailed Method for Titration

Using the pipette

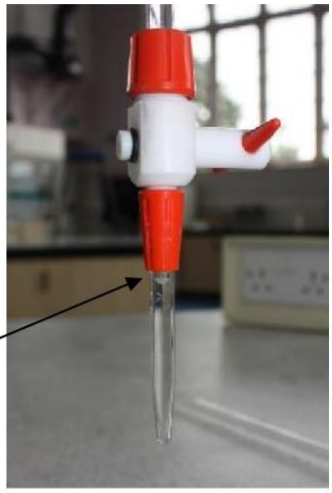
- **rinse p**ipette with substance to go in it (often alkali).
- **pipette 25 cm^3 of solution A into conical flask.** The volumetric pipette will have a mark on its neck to show the level to fill to. The bottom of the meniscus should sit on this line.
- **touch surface of solution with pipette** (to ensure correct amount is added). A small amount of solution will be left in the pipette at this stage. The calibration of the pipette will take into account this effect. It should not be forced out.

Make sure bottom of meniscus is on line on neck of pipette



Using the burette

The burette should be rinsed out with substance that will be put in it. If it is not rinsed out the acid or alkali added may be diluted by residual water in the burette or may react with substances left from a previous titration. This would lead to the concentration of the substance being lowered and a larger titre being delivered.



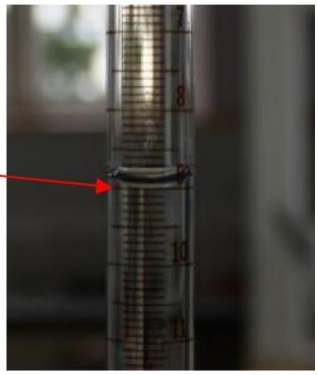
Don't leave the funnel in the burette because small drops of liquid may fall from the funnel during the titration leading to a false burette reading (would give a lower titre volume)

make sure the jet space in the burette **is filled** with the solution and air bubbles are removed.

If the jet space in the burette is not filled properly prior to commencing the titration it will lead to errors if it then fills during the titration, leading to a larger than expected titre reading.

Read the bottom of the meniscus on the burette

This is reading 9.00cm^3



Even though a burette has marking reading to 0.1cm^3, the burette readings should always be given to 2dp either ending in 0.0**0** or 0.0**5**. 0.0**5**cm^3 is the volume of 1 drop of solution delivered from a burette and so this is the smallest difference in readings that can be measured. If the bottom of the meniscus sits on a line it should end with a 0.0**0** as in the above example 9.0**0**cm^3. If the meniscus sits between two lines it should end 0.0**5**. e.g. if the bottom of the meniscus sits between the lines marked 9.1 and 9.2, you should record 9.1**5**

Adding indicator

Add **a few drops of indicator** and refer to colour change at end point

Indicators are generally weak acids so only add a few drops of them. If too much is added they will affect the titration result

phenolphthalein

If acid is added from the burette the colour change would be pink (alkali) to colourless (acid): end point pink colour just disappears [use with titrations using strong alkalis e.g. NaOH]

phenolphthalein
Alkali colour

phenolphthalein acid colour

Methyl orange

Methyl orange is a suitable indicator for neutralisation reactions where strong acids are used.
It is red in acid and yellow in alkali. It is orange at the end point.

Methyl orange
Alkali colour

Methyl orange
end point

Methyl orange
acid colour

Use a white tile underneath the flask to help observe the colour change

Add solution from burette whilst **swirling the mixture** and **add drop-wise at end point**

Distilled water can be added to the conical flask during a titration to wash the sides of the flask so that all the acid on the side is washed into the reaction mixture to react with the alkali.
It does not affect the titration reading as water does not react with the reagents or change the number of moles of acid added.

note burette reading before and after addition of solution
repeats titration until **at least 2 concordant results** are obtained- two readings within 0.1 of each other

A single titration could be flawed. Repeating allows for anomalous titres to be spotted and discounted

Only distilled water should be used to wash out conical flasks between titrations because it does not add any extra moles of reagents

If **2 or 3 values are within 0.10cm3** and therefore **concordant** or close then we can say results are accurate and **repeatable** and **the titration technique is good and consistent**

Recording results
- Results should be clearly recorded in a table
- Result should be recorded in full (i.e. both initial and final readings)
- **Record titre volumes to 2dp (0.05 cm3)**

Titration number	**1**	**2**	**3**
Initial burette reading (cm^3)	0.50	2.50	1.55
Final burette reading (cm^3)	24.50	27.00	25.95
Titre (cm^3)	24.00	24.50	24.40

Working out average titre results
Only make an average of the concordant titre results

Average titre = (24.50+ 24.40)/2
= 24.45

Safety precautions

Acids and alkalis are corrosive
(at low concentrations acids are irritants)
Wear eye protection and gloves
If spilled immediately wash affected parts after spillage

If substance is unknown treat it as potentially toxic and wear gloves.

Common Titration Equations

$CH_3CO_2H + NaOH \rightarrow CH_3CO_2^-Na^+ + H_2O$

$H_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2H_2O$

$HCl + NaOH \rightarrow NaCl + H_2O$

$NaHCO_3 + HCl \rightarrow NaCl + CO_2 + H_2O$

$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O$

Testing batches
In quality control it will be necessary to do titrations/testing on several samples as the amount/concentration of the chemical being tested may vary between samples.

Titrating mixtures
If titrating a mixture to work out the concentration of an active ingredient it is necessary to consider if the mixture contains other substances that have acid base properties.
If they don't have acid base properties we can titrate with confidence.

Uncertainty

Readings and Measurements

Readings
the values found from a single judgement when using a piece of equipment

Measurements
the values taken as the difference between the judgements of two values (e.g. using a burette in a titration)

In general, if uncertainty is not indicated on apparatus, the following assumptions are made:
For an analogue scale-
The uncertainty of a reading (one judgement) is at least ± 0.5 of the smallest scale reading.
The uncertainty of a measurement (two judgements) is at least ± 1 of the smallest scale reading.
- If the apparatus has a digital scale, the uncertainty is ± the resolution of the apparatus in each measurement

Calculating Apparatus Uncertainties

Each type of apparatus has a sensitivity uncertainty

- balance ± 0.001 g (if using a 3 d.p. balance)
- volumetric flask ± 0.1 cm^3
- 25 cm^3 pipette ± 0.1 cm^3
- burette (start & end readings and end point) ± 0.15 cm^3

Calculate the percentage error for each piece of equipment used by

$$\% \text{ uncertainty} = \frac{\pm \text{ uncertainty}}{\text{Measurement made on apparatus}} \times 100$$

e.g. for burette
% uncertainty = 0.15/average titre result x100

To calculate the maximum **total** percentage apparatus uncertainty in the final result add all the individual equipment uncertainties together.

Uncertainty of a measurement using a burette. If the burette used in the titration had an uncertainty for each reading of +/– 0.05 cm^3 then during a titration two readings would be taken so the uncertainty on the titre volume would be +/– 0.10 cm^3 .

Reducing uncertainties in a titration

Replacing measuring cylinders with pipettes or burettes which have lower apparatus uncertainty will lower the % uncertainty .

To reduce the % uncertainty in a burette reading it is necessary to make the titre a larger volume. This could be done by: increasing the volume and concentration of the substance in the conical flask or by decreasing the concentration of the substance in the burette.

To decrease the apparatus uncertainties you can either decrease the sensitivity uncertainty by using apparatus with a greater resolution (finer scale divisions) or you can increase the size of the measurement made.

If looking at a series of measurements in an investigation, the experiments with the smallest readings will have the highest experimental uncertainties.

Reducing uncertainties in measuring mass
Using a balance that measures to more decimal places or using a larger mass will reduce the % uncertainty in weighing a solid.

Weighing sample before and after addition and then calculating difference will ensure a more accurate measurement of the mass added.

Calculating the percentage difference between the actual value and the calculated value

If we calculated an Mr of 203 and the real value is 214, then the calculation is as follows:
Calculate difference 214-203 = 11
% = 11/214 x100
=5.41%

If the %**uncertainty** due to the apparatus **<** percentage difference between the actual value and the calculated value then there is a discrepancy in the result due to other errors.

If the %**uncertainty** due to the apparatus **>** percentage difference between the actual value and the calculated value then there is no discrepancy and all errors in the results can be explained by the sensitivity of the equipment.

Core practical 5: Investigation of the rates of hydrolysis of some halogenoalkanes

Testing for halogenoalkanes method

- Arrange three test tubes in a row and add three drops of halogenoalkane in the sequence 1-chlorobutane, 1-bromobutane, 1-iodobutane.
- Add 4 cm^3 of 0.02 M silver nitrate to each halogenoalkane.
- Without delay, put all three test tubes simultaneously in a hot water bath.
- Note the order in which precipitates appear

Comparing the rate of hydrolysis of halogenoalkanes reaction

Hydrolysis is defined as the splitting of a molecule (in this case a haloalkane) by a reaction with water

Water is a ***poor*** **nucleophile** but it can react ***slowly*** with haloalkanes in a substitution reaction

$$\mathbf{CH_3CH_2X + H_2O \rightarrow CH_3CH_2OH + X^- + H^+}$$

Aqueous silver nitrate is added to a haloalkane and the halide leaving group combines with a silver ion to form a **SILVER HALIDE PRECIPITATE**.
The precipitate only forms when the halide ion has left the haloalkane and so the rate of formation of the precipitate can be used to compare the reactivity of the different haloalkanes.

$CH_3CH_2\mathbf{I} + H_2O \rightarrow CH_3CH_2OH + \mathbf{I}^- + H^+$

$Ag^+_{(aq)} + \mathbf{I}^-_{(aq)} \rightarrow Ag\mathbf{I}_{(s)}$ - yellow precipitate

The iodoalkane forms a precipitate with the silver nitrate first as the C-I bond is weakest and so it hydrolyses the quickest

The **quicker** the **precipitate** is formed, the **faster** the **substitution** reaction and the **more reactive** the haloalkane

The rate of these substitution reactions depends on the strength of the C-X bond . The weaker the bond, the easier it is to break and the faster the reaction.

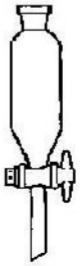
$Ag\mathbf{I}_{(s)}$ - yellow precipitate
$Ag\mathbf{Br}_{(s)}$ – cream precipitate forms faster
$Ag\mathbf{Cl}_{(s)}$ – white precipitate

Purifying an organic liquid

General method

- Put the distillate of impure product into a separating funnel
- wash product by adding either
  - sodium hydrogencarbonate solution , shaking and releasing the pressure from CO_2 produced.
  - Saturated sodium chloride solution
- Allow the layers to separate in the funnel, and then run and discard the aqueous layer.
- Run the organic layer into a clean, dry conical flask and add three spatula loads of drying agent (e.g. anhydrous sodium sulphate, calcium chloride) to dry the organic liquid. When dry the organic liquid should appear clear.
- Carefully decant the liquid into the distillation flask
- Distill to collect pure product

Separating funnel



Sodium hydrogencarbonate will neutralise any remaining reactant acid.

Sodium chloride will help separate the organic layer from the aqueous layer

The layer with lower density will be the upper layer. This is usually the organic layer

The drying agent should
- be insoluble in the organic liquid
- not react with the organic liquid

Decant means carefully pour off organic liquid leaving the drying agent in the conical flask

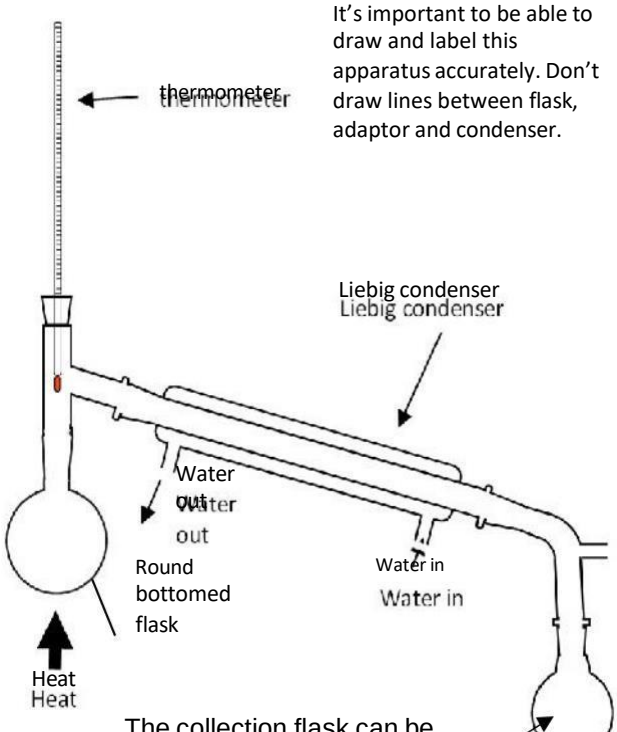
Distillation

In general used as separation technique to separate an organic product from its reacting mixture. Need to collect the distillate of the approximate boiling point range of the desired liquid.

Note the bulb of the thermometer should be at the T junction connecting to the condenser to measure the correct boiling point

Note the water goes in the bottom of the condenser to go against gravity. This allows more efficient cooling and prevents back flow of water.

Electric heaters are often used to heat organic chemicals. This is because organic chemicals are normally highly flammable and could set on fire with a naked flame.



It's important to be able to draw and label this apparatus accurately. Don't draw lines between flask, adaptor and condenser.

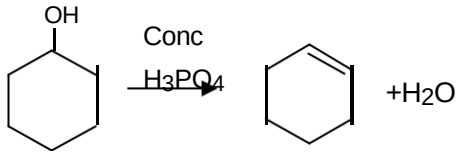
The collection flask can be cooled in ice to help improve yield of distillate

CORE PRACTICAL 6: Chlorination of 2-methylpropan-2-ol using concentrated hydrochloric acid

Detailed method for preparing and purifying a halogenoalkane from an alcohol

1. Measure 8 cm^3 of 2-methylpropan-2-ol in a measuring cylinder and measure its mass.
2. Pour the 2-methylpropan-2-ol into a separating funnel, and reweigh the measuring cylinder to find the mass of the 2-methylpropan-2-ol used.
3. In a fume cupboard, add 20 cm^3 of concentrated hydrochloric acid to the separating funnel, in portions of 3cm^3. After each portion, stopper the flask and invert it several times . Open the tap when doing this to release the pressure.
4. Allow the separating funnel to stand in the fume cupboard for about 20 minutes. Gently shake it at intervals.
5. After 20 minutes, allow the layers to separate in the funnel. Open the tap and remove the lower aqueous layer. Dispose of this layer.
6. Add sodium hydrogencarbonate solution in 2 cm^3 portions to the separating funnel. This neutralises any remaining acid. Shake the funnel after each addition, and release the pressure. Continue until no more bubbles of CO_2 are seen.
7. Allow the layers to separate in the funnel. Again remove and pour away the lower aqueous layer. Run off the organic layer into a clean conical flask and add two spatulas of anhydrous sodium sulfate. Stopper the flask, shake the contents and allow this to stand until the liquid becomes clear. This step dries the organic liquid.
8. Decant the liquid into a weighed clean distillation flask.
9. Distil the liquid by holding a 250ml beaker half-full of boiled water around the flask using standard distillation set up. Collect the liquid that distils in the range 47-53^oC.
10. Measure the mass of the 2-chloro-2-methylpropane collected.

Detailed method for preparing and purifying Cyclohexene from cyclohexanol



a) Pour about 20 cm^3 of cyclohexanol into a weighed 50 cm^3 pear-shaped flask. Reweigh the flask and record the mass of cyclohexanol.
b) Using a plastic graduated dropping pipette, carefully and with frequent shaking, add to the flask approximately 8 cm^3 of concentrated phosphoric acid.
c) Add a few anti-bumping granules to the flask and assemble the distillation apparatus, so that the contents of the flask may be distilled. Heat the flask gently, distilling over any liquid which boils below 100 °C.
d) Pour the distillate into a separating funnel and add 50 cm^3 of saturated sodium chloride solution. Shake the mixture and allow the two layers to separate.
e) run off the lower layer into a beaker and then transfer the upper layer, which contains the crude cyclohexene, into a small conical flask.
f) Add a few lumps of anhydrous calcium chloride or anhydrous sodium sulfate(VI) or anhydrous magnesium sulfate to the crude cyclohexene to remove water. Stopper the flask, shake the contents and allow this to stand until the liquid becomes clear.
g) Decant the liquid into a clean, dry, weighed sample container.
h) Reweigh the container, calculate the mass of dry cyclohexene produced

Detailed Method for Preparing and Purifying an Ester

Propyl ethanoate can be made in the laboratory from propan-1-ol and ethanoic acid.

The equation for the reaction is

$CH_3COOH + CH_3CH_2CH_2OH \rightarrow CH_3COOCH_2CH_2CH_3 + H_2O$

Procedure

1. Propan-1-ol (50 cm^3) and ethanoic acid (50 cm^3) are mixed thoroughly in a 250 cm^3 round-bottomed flask.
2. **Concentrated sulfuric acid** (10 cm_3) is added **drop by drop to the mixture**, keeping the contents of the flask well-shaken and **cooled** in an ice-water bath.

Sulfuric acid is a catalyst

Adding conc H_2SO_4 is an exothermic reaction- to prevent uncontrolled boiling over add **drop by drop** and **cool**

3. When the acid has all been added, a **reflux condenser** is fitted to the flask and the mixture gently boiled over an **electric heating mantle** for about **30 minutes.**

In **reflux t**he reactant vapours of volatile compound are condensed and returned to the reaction mixture. The reaction is slow so it is heated for **30 minutes The electric heating mantle** allows for controlled heating and stops flammable vapour lighting

4. The mixture is cooled, and the apparatus rearranged for distillation. The crude ester (about 60 cm^3) is distilled off.
5. The distillate is placed in a separating funnel and shaken with about half its volume **of 30% sodium carbonate** solution, with the **pressure being released at intervals**. The *lower* **aqueous layer is then discarded**.

Sodium carbonate reacts with unreacted acid and remaining catalyst still present after distillation. The reaction produces CO_2 so the **pressure** of gas needs to be **released.**
The upper layer is organic because it has a lower density than water

6. The crude ester is shaken in a separating funnel with about half its volume of 50% calcium chloride solution, which removes unreacted alcohol. The lower layer is discarded.

7. The ester is run into a clean, dry flask containing **some anhydrous calcium chloride** and swirled.

Calcium chloride is a drying agent. The liquid will appear clear when dry.

8. The ester is filtered into a clean, dry flask, with a **few anti-bumping granules**, and distilled. The fraction boiling between 100°C and 103°C is collected.

Anti-bumping granules are added to the prevent vigorous, uneven boiling by making small bubbles form instead of large bubbles

Measuring boiling point

Purity of liquid can be determined by measuring a boiling point. This can be done in a distillation set up or by simply boiling a tube of the sample in an heating oil bath. If the liquid is pure it will have the boiling point referred to in data books. If impure the boiling point tends to be higher than the pure liquid

To get a correct measure of boiling point the thermometer should be above the level of the surface of the boiling liquid and be measuring the temperature of the saturated vapour.

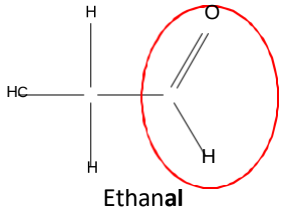
Pressure should be noted as changing pressure can change the boiling point of a liquid

Measuring boiling point is not the most accurate method of identifying a substance as several substances may have the same boiling point.

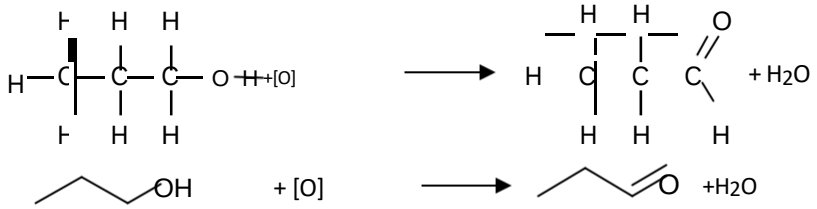
Core practical 7. Oxidation of an alcohol

Partial Oxidation of Primary Alcohols

Reaction: primary alcohol → aldehyde
Reagent: potassium dichromate (VI) solution and dilute sulfuric acid.
Conditions: (use a limited amount of dichromate) warm gently and **distil** out the aldehyde as it forms:



Ethanal



$CH_3CH_2CH_2OH + [O] \rightarrow CH_3CH_2CHO + H_2O$

propan-1-ol **propanal**

Observation: the orange dichromate ion ($Cr_2O_7^{2-}$) reduces to the green Cr^{3+} ion

SAFETY
You must wear gloves when handling solid potassium dichromate(VI) since it is highly toxic and a category 2 carcinogen; it is also an irritant. Avoid inhaling any dust.
Concentrated sulfuric acid is corrosive.

Detailed Method: The partial oxidation of propan-1-ol

This experiment uses a limited quantity of oxidising agent (0.01 mol) and the product is distilled from the reaction mixture immediately it is formed. In this way we hope to achieve a partial oxidation of propan-1-ol.

- Place about 10 cm^3 of dilute sulfuric acid in a flask and add about 3g of potassium dichromate(VI) and 2 or 3 anti-bumping granules. Shake the contents of the flask until solution is complete (do not warm).
- Add 1.5 cm^3 of propan-1-ol in drops from a dropping pipette, shaking the flask so as to mix the contents, and then assemble distillation apparatus as shown below
- Gently heat and slowly distil 2 cm^3 of liquid into a test tube, taking care that none of the reaction mixture splashes over.

Distillation

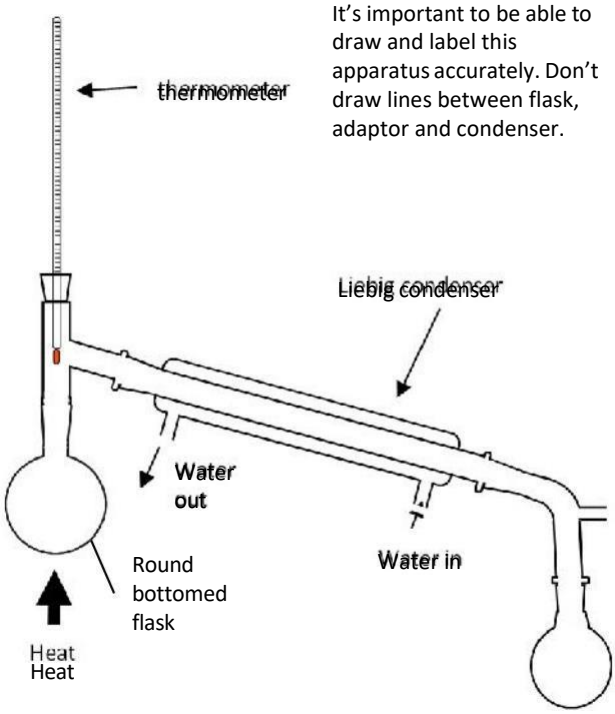
In general used as separation technique to separate an organic product from its reacting mixture. Need to collect the distillate of the approximate boiling point range of the desired liquid.

Note the bulb of the thermometer should be at the T junction connecting to the condenser to measure the correct boiling point

Note the water goes in the bottom of the condenser to go against gravity. This allows more efficient cooling and prevents back flow of water.

Electric heaters are often used to heat organic

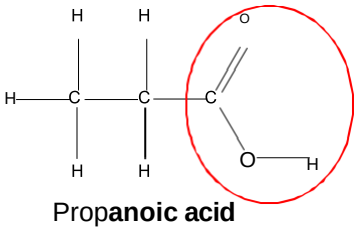
chemicals. This is because organic chemicals are normally highly flammable and could set on fire with a naked flame.



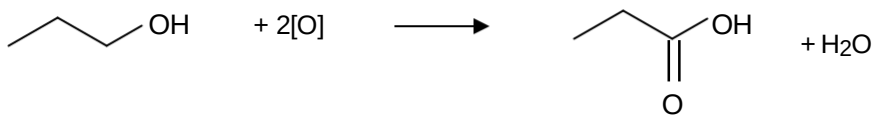
It's important to be able to draw and label this apparatus accurately. Don't draw lines between flask, adaptor and condenser.

Reflux: Full Oxidation of Primary Alcohols

Reaction: primary alcohol → carboxylic acid
Reagent: potassium/sodium dichromate(VI) solution and sulfuric acid
Conditions: use an excess of dichromate, and **heat under reflux**: (distil off product after the reaction has finished)



Propanoic acid



$CH_3CH_2CH_2OH + 2[O] \rightarrow CH_3CH_2COOH + H_2O$

propan-1-ol **Propanoic acid**

Observation: the orange dichromate ion ($Cr_2O_7^{2-}$) reduces to the green Cr^{3+} ion

Detailed method

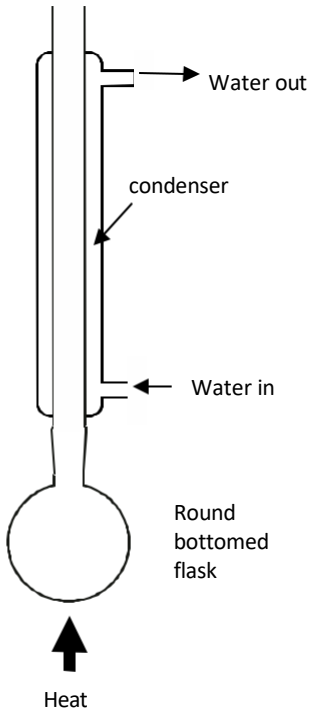
- Measure 5 cm^3 of water into a boiling tube. Add 6 g of sodium dichromate(VI), shake and set aside to dissolve.
- Put about 1.5 cm^3 propan-1-ol into a 50 cm^3 round bottomed flask and add about 5 cm^3 of water and two or three anti-bumping granules. Put a condenser on the flask for **reflux**, as shown in figure below.
- Add 2 cm^3 of concentrated sulfuric acid down the condenser in drops from a dropping pipette. While the mixture is still warm, start to add your sodium dichromate(VI) solution down the condenser in drops from a dropping pipette. The energy released from the reaction should make the mixture boil. Add the solution a drop at a time so that the mixture continues to boil without any external heating.
- When all the sodium dichromate(VI) solution has been added, use a low Bunsen burner flame to keep the mixture boiling for 10 minutes, not allowing any vapour to escape.
- At the end of that time remove the Bunsen burner and arrange the apparatus for distillation. Gently distil 2-3 cm^3 of liquid into a test tube.

Reflux

Reflux is used when heating organic reaction mixtures for long periods. The condenser prevents organic vapours from escaping by condensing them back to liquids. The reactant vapours of volatile compound are condensed and returned to the reaction mixture.

Never seal the end of the condenser as the build up of gas pressure could cause the apparatus to explode. This is true of any apparatus where volatile liquids are heated including the distillation set up

Anti -bumping granules are added to the flask in both distillation and reflux to prevent vigorous, uneven boiling by **making small bubbles** form instead of large bubbles



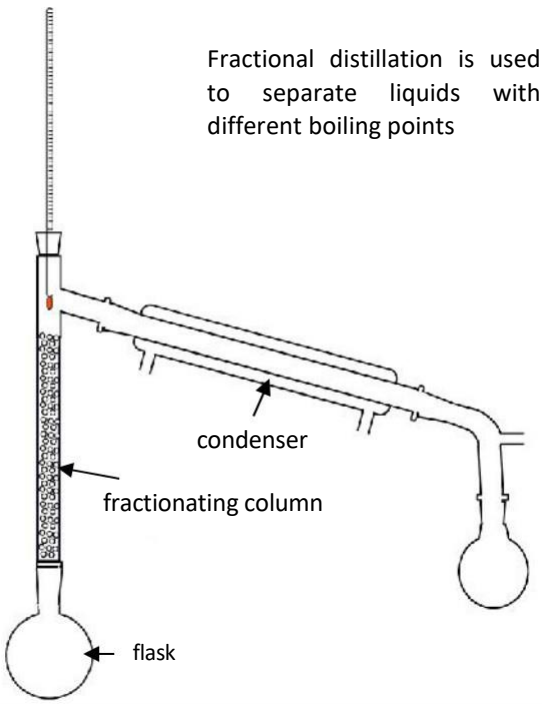
It's important to be able to draw and label this apparatus accurately.

- Don't draw lines between flask and condenser.
- Don't have top of condenser sealed
- Condenser must have outer tube for water that is sealed at top and bottom
- Condenser must have two openings for water in and out that are open

Fractional Distillation: In the laboratory

- Heat the flask, with a Bunsen burner or electric mantle
- This causes vapours of all the components in the mixture to be produced.
- Vapours pass up the fractionating column.
- The vapour of the substance with the lower boiling point reaches the top of the fractionating column first.
- The thermometer should be at or below the boiling point of the most volatile substance.
- The vapours with higher boiling points condense back into the flask.
- Only the most volatile vapour passes into the condenser.
- The condenser cools the vapours and condenses to a liquid and is collected.

Fractional distillation is used to separate liquids with different boiling points



CORE PRACTICAL 8 + 15: Analysis of some inorganic unknowns

Testing for cations

Flame tests

Method
Use a **nichrome wire** (nichrome is an unreactive metal and will not give out any flame colour)
Clean the wire by dipping **in concentrated hydrochloric acid** and then heating in Bunsen flame
If the sample is not powdered then grind it up.
Dip wire in solid and put in Bunsen flame and observe flame

Lithium : Scarlet red
Sodium : Yellow
Potassium : lilac
Rubidium : red
Caesium: blue
Magnesium: no flame colour (energy emitted of a wavelength outside visible spectrum)
Calcium: brick red
Strontium: red
Barium: apple green

Method: adding dilute sodium hydroxide

a) Place about 10 drops of 0.1 mol dm^{-3} metal ion solution in a test tube.
b) Add about 10 drops of 0.6 mol dm^{-3} sodium hydroxide solution, mixing well.
c) Continue to add sodium hydroxide solution, dropwise with gentle shaking, until in excess

This test can be used on group 2 metal ions and transition metal ions.

Results for Group 2

The results in this test are an application of the trend that group II hydroxides become **more soluble** down the group.

Magnesium hydroxide is classed as insoluble in water and will appear as **a white precipitate**.

Simplest Ionic Equation for formation of $Mg(OH)_2$ (s)

$$Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s).$$

A suspension of magnesium hydroxide in water will appear slightly alkaline (pH 9) so some hydroxide ions must therefore have been produced by a very slight dissolving.

Calcium hydroxide is classed as partially soluble in water and will appear as **a white precipitate (**it may need more sodium hydroxide to be added before it appears compared to a magnesium solution.)

Simplest Ionic Equation for formation of $Ca(OH)_2$ (s)

$$Ca^{2+}(aq) + 2OH^-(aq) \rightarrow Ca(OH)_2(s).$$

A suspension of calcium hydroxide in water will appear more alkaline (pH 11) than magnesium hydroxide as it is more soluble so there will be more hydroxide ions present in solution.

Strontium and **barium** salts will not form a hydroxide precipitate on addition of sodium hydroxide due to their high solubility. The solutions will be highly alkaline

Testing for Ammonium ions (NH_4^+)

a) Place about 10 drops of 0.1 mol dm^{-3} ammonium chloride in a test tube.
b) Add about 10 drops of 0.4 mol dm^{-3} sodium hydroxide solution. Shake the mixture.
c) Warm the mixture in the test tube gently using a water bath.
d) Test the fumes released from the mixture by holding a piece of damp red litmus paper in the mouth of the test tube.

Results: alkaline ammonia gas is released which turns the red litmus paper blue

CORE PRACTICAL 8+15: Analysis of some inorganic unknowns

Testing for anions: – Group 7 (halide ions), OH^- , CO_3^{2-} , SO_4^{2-}

Testing for presence of a sulfate ion

$BaCl_2$ solution acidified with hydrochloric acid is used as a reagent to test for sulphate ions.

If acidified **Barium Chloride** is added to a solution that contains sulfate ions a **white precipitate** of Barium Sulfate forms.

Simplest ionic equation
$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$.

Other anions should give a negative result which is no precipitate forming.

The hydrochloric acid is needed to react with carbonate impurities that are often found in salts which would form a white Barium carbonate precipitate and so give a false result. You could not used sulphuric acid because it contains sulphate ions and so would give a false positive result.

$2HCl + Na_2CO_3 \rightarrow 2NaCl + H_2O + CO_2$

Fizzing due to CO_2 would be observed if a carbonate was present.

Testing for presence of halide ions with silver nitrate.

This reaction is used as a test to identify which halide ion is present. The test solution is made acidic with **nitric acid,** and then **Silver nitrate solution** is added dropwise.

The role of nitric acid is to react with any carbonates present to prevent formation of the precipitate Ag_2CO_3. This would mask the desired observations

$2 HNO_3 + Na_2CO_3 \rightarrow 2 NaNO_3 + H_2O + CO_2$

Fluorides produce no precipitate
Chlorides produce a **white precipitate**
$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$
Bromides produce a **cream precipitate**
$Ag^+(aq) + Br^-(aq) \rightarrow AgBr(s)$
Iodides produce a **pale yellow precipitate**
$Ag^+(aq) + I^-(aq) \rightarrow AgI(s)$

The silver halide precipitates can be treated with ammonia solution to help differentiate between them if the colours look similar:

Silver chloride dissolves in **dilute ammonia** to form a complex ion
$AgCl(s) + 2NH_3(aq) \rightarrow [Ag(NH_3)_2]^+(aq) + Cl^-(aq)$
Colourless solution
Silver bromide dissolves in **concentrated ammonia** to form a complex ion
$AgBr(s) + 2NH_3(aq) \rightarrow [Ag(NH_3)_2]^+(aq) + Br^-(aq)$
Colourless solution
Silver iodide does not react with ammonia – it is too insoluble.

Testing for presence of carbonate ions

Add any dilute acid and observe effervescence.

Bubble gas through limewater to test for CO_2 – will turn limewater cloudy $2HCl + Na_2CO_3 \rightarrow 2NaCl + H_2O + CO_2$

Fizzing due to CO_2 would be observed if a carbonate was present

Testing for presence of a hydroxide ions

Alkaline hydroxide ions will turn red litmus paper blue

Reactions of halide salts with concentrated sulfuric acid.

The halides show increasing power as reducing agents as one goes down the group. This can be clearly demonstrated in the various reactions of the solid halides with concentrated sulfuric acid.

Know the equations and observations of these reactions very well.

Explanation of differing reducing power of halides A reducing agent donates electrons.
The reducing power of the halides increases down group 7 They have a greater tendency to donate electrons.
This is because as the ions get bigger it is easier for the outer electrons to be given away as the pull from the nucleus on them becomes smaller.

Fluoride and Chloride

F^- and Cl^- ions are not strong enough reducing agents to reduce the S in H_2SO_4. No redox reactions occur. Only acid-base reactions occur.

$NaF(s) + H_2SO_4(l) \rightarrow NaHSO_4(s) + HF(g)$ Observations:

White steamy fumes of HF are evolved. $NaCl(s) + H_2SO_4(l) \rightarrow NaHSO_4(s) + HCl(g)$ Observations: White steamy fumes of HCl are evolved.

These are acid –base reactions and not redox reactions. H_2SO_4 plays the role of an acid (proton donor).

Bromide

Br- ions are stronger reducing agents than Cl- and F- and after the initial acid-base reaction reduce the sulfur in H_2SO_4 from +6 to + 4 in SO_2

Acid- base step: $NaBr(s) + H_2SO_4(l) \rightarrow NaHSO_4(s) + HBr(g)$

Redox step: $2HBr + H_2SO_4 \rightarrow Br_2(g) + SO_2(g) + 2H_2O(l)$

Observations: White steamy fumes of HBr are evolved.

Red fumes of Bromine are also evolved and a colourless, acidic gas **SO_2**

Ox ½ equation $2Br^- \rightarrow Br_2 + 2e^-$

Re ½ equation $H_2SO_4 + 2H^+ + 2e^- \rightarrow SO_2 + 2H_2O$

Reduction product = sulfur dioxide

Note the H_2SO_4 plays the role of acid in the first step producing HBr and then acts as an oxidising agent in the second redox step.

Iodide

I- ions are the strongest halide reducing agents. They can reduce the sulfur from +6 in H_2SO_4 to + 4 in SO_2, to 0 in S and -2 in H_2S.

$NaI(s) + H_2SO_4(l) \rightarrow NaHSO_4(s) + HI(g)$
$2HI + H_2SO_4 \rightarrow I_2(s) + SO_2(g) + 2H_2O(l)$
$6HI + H_2SO_4 \rightarrow 3I_2 + S(s) + 4H_2O(l)$
$8HI + H_2SO_4 \rightarrow 4I_2(s) + H_2S(g) + 4H_2O(l)$

Observations:
White **steamy fumes** of HI are evolved.
Black solid and **purple fumes** of Iodine are also evolved
A **colourless**, acidic **gas** SO_2
A **yellow solid** of sulfur
H_2S (Hydrogen sulfide), a **gas** with a **bad egg smell.**

Ox ½ equation $2I^- \rightarrow I_2 + 2e^-$

Re ½ equation $H_2SO_4 + 2H^+ + 2e^- \rightarrow SO_2 + 2H_2O$

Re ½ equation $H_2SO_4 + 6H^+ + 6e^- \rightarrow S + 4H_2O$ Re ½ equation $H_2SO_4 + 8H^+ + 8e^- \rightarrow H_2S + 4H_2O$

Reduction products = sulfur dioxide, sulfur and hydrogen sulfide

Note the H_2SO_4 plays the role of acid in the first step producing HI and then acts as an oxidising agent in the three redox steps

Often in exam questions these redox reactions are worked out after first making the half-equations

More on Insoluble salts and Precipitation reactions

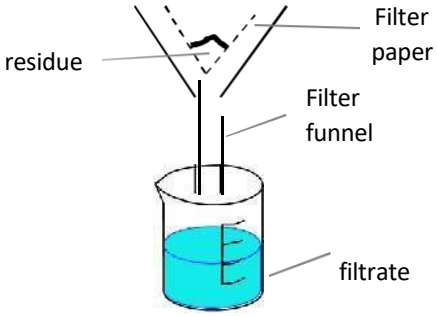
Insoluble salts can be made by mixing appropriate solutions of ions so that a **precipitate** is formed Barium nitrate (aq) + sodium sulfate (aq) → **barium sulfate (s)** + sodium nitrate (aq)
These are called **precipitation** reactions. A **precipitate is a solid**

There are some common rules for solubility of salts. No syllabus requires these to be learnt but a good chemist does know them.

Soluble salts	Insoluble salts
All sodium, potassium and ammonium salts	
All nitrates	
Most chlorides, bromides, iodides	Silver, lead chlorides, bromides iodides
Most sulfates	Lead, strontium and barium sulfate
Sodium, potassium and ammonium carbonates	Most other carbonates
Sodium, potassium and ammonium hydroxides	Most other hydroxides

When making an insoluble salt, normally the salt would be removed by **filtration,** washed with **distilled water to remove soluble impurities** and then **dried on filter paper**

Filtration



This is gravitational filtration. Use if small amounts of solid are formed.

Writing Ionic equations for precipitation reactions

We usually write ionic equations to show precipitation reactions. Ionic equations only show the ions that are reacting and leave out spectator ions.

Spectator ions are ions that are
- Not changing state
- Not changing oxidation number

Take full equation

$$Ba(NO_3)_2\ (aq) + Na_2SO_4\ (aq) \rightarrow BaSO_4\ (s) + 2\ NaNO_3\ (aq)$$

Separate (aq) solutions into ions

$$Ba^{2+}_{(aq)} + 2NO_3^-(aq) + 2Na^+(aq) + SO_4^{2-}(aq) \rightarrow BaSO_{4(s)} + 2\ Na^+_{(aq)} + 2NO_3^-(aq)$$

Cancel out spectator ions leaving the simplest ionic equation

$$Ba^{2+}\ (aq) + SO_4^{2-}(aq) \rightarrow BaSO_4\ (s).$$

CORE PRACTICAL 8+15: Analysis of some organic unknowns

Tests for alcohol, aldehyde, alkene and carboxylic acid

Functional group test for an Alkene

To 0.5 cm^3 of bromine water in a test tube add a few drops of the unknown and shake.

Observation: alkenes should decolourise bromine water

Fehling's solution

Reagent: Fehling's solution containing blue Cu^{2+} ions.

Conditions: heat gently

Reaction: aldehydes only are oxidised by Fehling's Solution into a carboxylic acid and the copper (II) ions are reduced to copper(I) oxide .

Observation: Aldehydes : Blue Cu^{2+} ions in solution change to a red precipitate of Cu_2O. **Ketones do not react**

$CH_3CHO + 2Cu^{2+} + 2H_2O \rightarrow CH_3COOH + Cu_2O + 4H^+$

Fehling's solution method

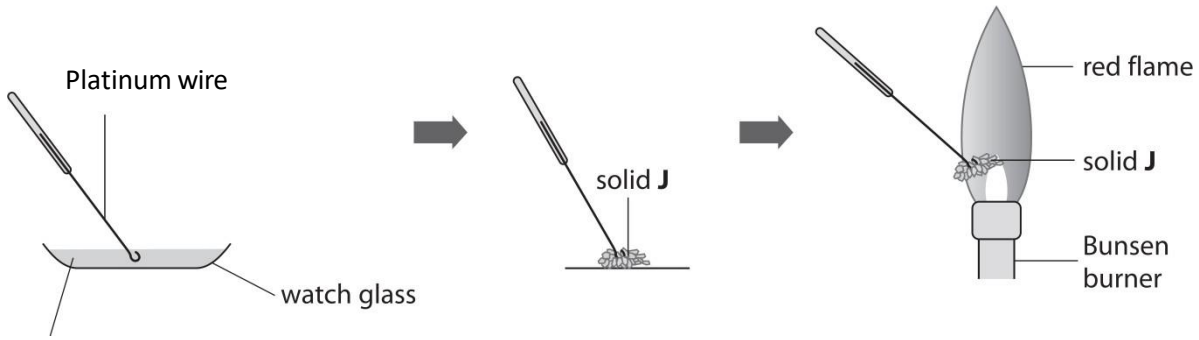
Place 1 cm^3 of Fehling's A into each of two boiling tubes, and then add Fehling's B until the blue precipitate redissolves. Add a few drops of the unknown and leave in the water bath for a few minutes.

Summary of Identification of Functional Groups by test-tube reactions

Functional group	Reagent	Result
Alkene	Bromine water	Orange colour decolourises
Alcohols	PCl_5	Misty fumes of HCl produced
Alcohols	Sodium metal	Efferevesence due to H_2 gas
Aldehyde	Fehling's solution	Blue solution to red precipitate
1^o 2^o alcohol and aldehyde	Sodium dichromate and sulfuric acid	Orange to green colour change
chloroalkane	Warm with silver nitrate	Slow formation of white precipitate of AgCl

Carrying out flame test

- Use a clean Nichrome wire or platinum wire.
- Dip nichrome wire into **CONCENTRATED** hydrochloric acid and then into the salt.
- The wire is held in progressively hotter parts of a non-luminous Bunsen flame.



Flame tests

FLAME COLOUR	INFERENCE
yellow	Sodium ion
Lilac	Potassium ion
Yellow-red	Calcium ion
Red	Lithium
Red	Strontium
Pale green	barium

Note: Further tests would be needed to distinguish between Li, Sr and Ca

Action of heat on solids – gases or vapour maybe evolved when a solid compound is heated

GAS OR VAPOUR	POSSIBLE SOURCE
Carbon dioxide	Carbonates of metals other than group 1
Oxygen	Group 1 nitrates (other than Li)
Oxygen and nitrogen dioxide	Nitrates (other than Na and K)
water	Hydrated salts

Recognition and Identification of common gases

GAS	TEST	OBSERVATION
Oxygen (O_2)	Insert a glowing splint into the gas	The splint relights/rekindles
Carbon dioxide (CO_2)	Bubble gas through lime water	The lime water turns milky/forms white ppt
Ammonia (NH_3)	Insert a piece of moist red litmus paper into the gas	The moist red litmus paper turn blue
	Dip a glass rod in dilute hydrochloric acid and insert it into the gas	Dense white fumes are formed
Nitrogen dioxide (NO_2)	Appearance or colour of gas	Brown gas (insoluble in organic solvents)
	Add starch - iodide paper	Gas turns starch iodide paper blue black
Hydrogen (H_2)	Insert a lighted/burning splint into the gas	Colourless gas which ignites with a "pop" OR burning splint goes off with a "pop" sound
Hydrogen chloride (HCl)	Dip a glass rod in aqueous ammonia and insert into the gas	Dense white fumes/smoke formed
	Test with blue litmus paper	The steamy fumes produced changes blue litmus to red
Chlorine (Cl_2)	Insert a piece of moist blue litmus paper into the gas	The pale green gas turns moist blue litmus paper to red and then bleaches it
Bromine (Br_2)	Appearance or colour of gas	Brown gas that dissolves in organic solvents
Iodine (I_2)	Appearance or colour of gas	Purple vapour
Water vapour (H_2O)	Insert a piece of blue anhydrous cobalt(II) chloride paper into the gas	The blue cobalt chloride paper turns pink
Sulphur dioxide (SO_2)	Pass the gas through acidified potassium dichromate (VI) solution	Orange colour turns to green

Tests for anions

ANION	TEST	OBSERVATION
Carbonate , CO_3^{2-}	Add aqueous acid to the solid carbonate oraq carbonate solution (eg. HCl or H_2SO_4)	Effervescence observed. A colourless gas which turns lime water milky is given off.
	Heat the solid carbonate	Carbonates other than Na, K and Ba give off carbondioxide
	Add magnesium suphate ($MgSO_4$)	A white precipitate is formed at room temperature
Hydrogen carbonate, HCO_3^-	Add solid HCO_3^- to almost boiling water	Effervescence observed. Colourless gas given off turns lime water milky.
	Add magnesium suphate ($MgSO_4$)	No precipitate formed at room temperature. A white precipitate is formed on heating.
Sulphate, SO_4^{2-}	Add barium chloride ($BaCl_2$) followed by dilute HClacid	A white precipitate ($BaSO_4$) is formed which is insoluble in excess dilute HClacid
Sulphite, SO_3^{2-}	Add barium chloride ($BaCl_2$) followed by dilute HCl	A white precipitate ($BaSO_3$) is formed which is soluble in excess dilute HCl acid
	Add dilute $H_2SO_4$to the solid or solution and warm. Test the gas given off by passing it into acidified $K_2Cr_2O_7$ solution	The orange solution turns green
Nitrate, NO_3^-	Add aqueous sodium hydroxide (NaOH) and Al (or Zinc powder), warm. Test gas given off using damp red litmus paper	A pungent smelling gas that turns red litmus paper to blue is given off

Tests for cations

CATION	TEST	OBSERVATION
NH_4^+, ammonium ion	Add aqueous sodium hydroxide to solid or aq solution and warm. Test gas given off with damp red litmus paper	Pungent smelling gas (ammonia) given off which turns damp red litmus paper blue
Na^+	Flame test	Yellow
K^+	Flame test	Lilac
Mg^{2+}	Add dilute aqueous ammonia to a solution containing magnesium ions	A white precipitate is formed
	Add ammonium carbonate solution	A white precipitate(of magnesium carbonate) is formed
Ca^{2+}	Flame test	Red
	Add concentrated NaOH	A white precipitate is formed
Ba^{2+}	Flame test	Pale green
Li^+	Flame test	Crimson Red
Sr^{2+}	Flame test	Red

Tests for halide ions in aqueous solution

(1) **Test with $AgNO_3$ and HNO_3**: To the test solution add dilute nitric acid followed by silver nitrate solution. Test the solubility of precipitate formed by adding dilute aqueous ammonia and concentrated aqueous ammonia.

HALIDE ION	OBSERVATION
Cl^-	White precipitate formed, which is soluble in dilute and concentrated aq. ammonia
Br^-	Cream precipitate formed, which is insoluble in dilute aq. Ammonia but soluble in concentrated aq. ammonia

I^-	A pale yellow precipitate formed, which is insoluble in dilute and concentrated aq. ammonia

(2) **Test with $Pb(NO_3)_2$**: To the test solution add few drops of lead (II) nitrate solution. Heat the precipitate and allow the liquid to cool

HALIDE ION	OBSERVATION
Cl^-	White precipitate formed dissolves on heating. White crystals reappear on cooling
Br^-	Whiteprecipitate formed dissolves on heating. White crystals reappear on cooling
I^-	Yellow precipitate formed dissolved on heating. Golden or yellow crystals reappear on cooling

(3) **Chlorine water test**: To the test solution add few drops of chlorine water followed by about 2.00cm^3 of organic solvent (eg: Hexane, CCl_4, $CHCl_3$). Shake the mixture well.

HALIDE ION	OBSERVATION
Cl^-	No change when shaken with organic solvent $Cl_2(aq) + Cl^-(aq) \rightarrow$ No Reaction
Br^-	The organic layer turns reddish brown in colour $Cl_2(aq) + 2Br^-(aq) \rightarrow Br_2(aq) + 2Cl^-(aq)$
I^-	The organic layer turns purple or violet in colour. Aqueous layer turns brown $Cl_2(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2Cl^-(aq)$

Tests for halide salts in solid state

To the solid halide add concentrated sulphuric acid.

SOLID SODIUM HALIDE	OBSERVATION	INFERENCE
NaCl	White misty fumes	HCl
NaBr	White misty fumes Brown fumes or vapour Colourless gas with choking odour	HBr Br_2 SO_2
NaI	White misty fumes Purple vapour and a black precipitate	HI

	Rotten egg smelling colourless gas Colourless gas with choking smell Yellow precipitate	I_2 H_2S SO_2 S

Tests for halogens

TEST	Cl_2	Br_2	I_2
Effect on damp red litmus paper	Bleach rapidly	Bleach slowly than chlorine	No effect
Effect on damp blue litmus paper	Turns red and bleaches it	Turns red and bleaches it, slower than with Cl_2	No effect
Add KBr followed by an organic solvent (such as hexane) and shake well	A brown solution is formed. On adding hexane the organic layer turns reddish brown in colour	No effect	No effect
Add KI followed by hexane. Shake the mixture	A brown solution is formed. On shaking with hexane, the organic layer turns violet in colour	A brown solution is formed. On shaking with hexane, the organic layer turns violet in colour	No effect
Add starch solution	No effect	No effect	Turns blue-black colour

Lab apparatus for unit 3

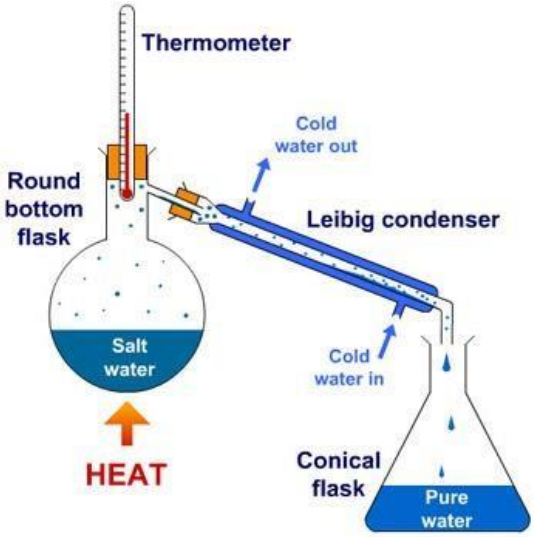
Distillation

Distillation, process used to separate the substances composing a mixture of miscible liquids of varying boiling points

Distillation is a complicated method that really will only work with the right equipment.

Method:

1. Pour your solution into a flask , then place the flask onto Bunsen Burner and heat.
2. The liquid with the lowest boiling point will evaporate.
3. The vapor will then pass through the Liebig Condenser.
4. The Condenser has 2 pipes, the vapor will pass through the center one.
5. The outer pipe will have water running through it.
6. The vapor will condense and then drip into a beaker.

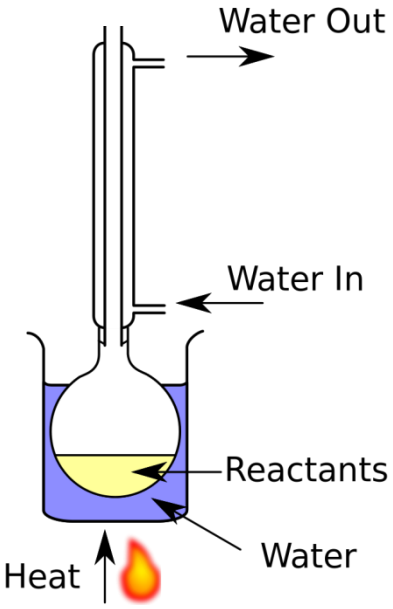


Question: Explain how the Liebig condenser works and its purpose in distillation.

- Cold **water** passing through the condenser cools and condenses the vapours or gases rising from the reaction mixture.
- It prevents escape of reactants and products so that the reaction can continue to completion.

Heat under reflux

Heating under reflux enables a mixture including volatile materials to be heated for a long time without loss of solvent.



Notes:

- The water goes in at the bottom of the condenser
- There must not be a stopper in the top of the condenser - the apparatus **must not be sealed**.
- There **must not be a thermometer** in the top of the condenser, partly for the same reason as above but also because it wouldn't measure the temperature of anything that mattered. The vapour doesn't get that far.

Separating Funnel

A separating funnel is used to separate two liquids with different densities

1. Get a immiscible solution e.g. oil and water
2. The liquids are placed in the separating filter, they form two layers
3. Then you slowly turn the tap,so that all the water (which is denser than oil)trickles out
4. When all the water is gone you close the tap.

It is not perfect as you can never be sure were one liquid ends and the other begin.

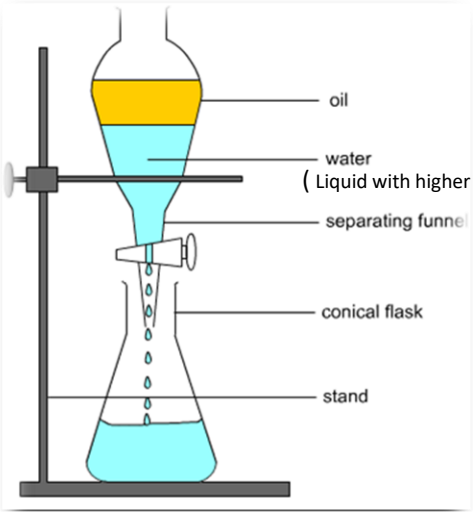
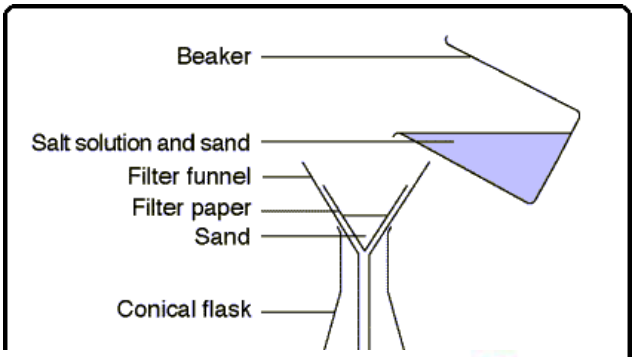


Diagram of Apparatus

Filtration

A method to separate an insoluble solid is to pour the mixture onto filter paper in a filter funnel.
The filter paper will absorb the liquid, which will then drip through the funnel
The solid sediment will be left behind

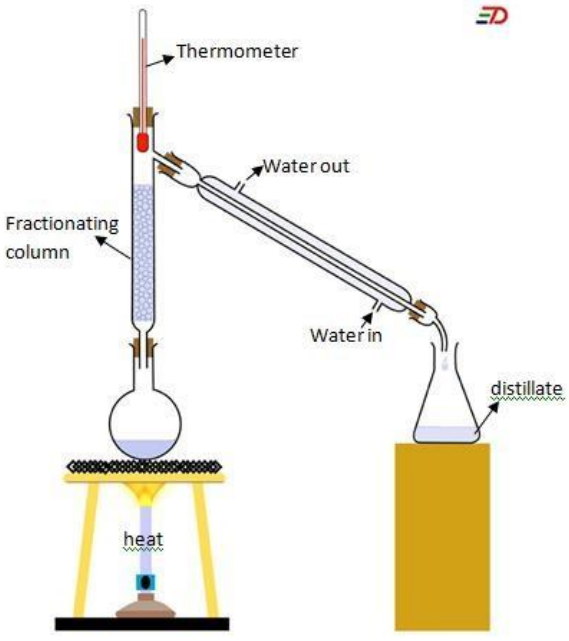


Method:

1. Fold the filter paper into quarters.
2. Fit it into the funnel.
3. Slowly pour the liquid and sediment.
4. Liquid will drip through the funnel.
5. Sediment will be left behindAnd you are left with a pure liquid.

Fractional distillation

Fractional distillation is the separation of a liquid mixture into fractions differing in boiling point by means of distillation, typically using a fractionating column.



Energetics

To determine the enthalpy change of combustion